2690

Effect of Oxygen Removal Technique on Flavor Stability of Low-Heat Foam Spray Dried Whole Milk

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Abstract

Oxygen-removal techniques, before gas packing, have been examined with the view of stabilizing the flavor of low-heat foam spray dried whole milk during storage. Holding 18 hr under a pressure of 1 mm, then packing in nitrogen ($O_2 < 0.002\%$), reduced but did not completely eliminate development of oxidized flavor in the product during six months of storage at 4 C. This treatment was effective for stabilizing the flavor of foam vacuum dried whole milk. Oxidized flavor development in stored foam spray dried whole milk was eliminated by packing the powder in cans containing a noble metal catalyst pellet plus nitrogen containing 5% hydrogen. The catalyzed reaction between hydrogen and oxygen to form water rapidly reduced the oxygen in the packs to 0.001% or less. Holding the powder 15 min at 1 mm pressure before filling cans with the $N_2 + H_2$ mixture was sufficient to effectively pack both types of foam dried material. Difficulties encountered in producing low oxygen content packs apparently relate to milk powder particle structure.

It has been found virtually impossible to emove all of the oxygen from spray dried milk y vacuum treatment prior to gas packing (3). 'his was ascribed to structural properties of pray dried milk which prevented rapid renoval of air from the interior of the powder :ranules. The implication that the air trapped n the interior of the particles would result n poor keeping quality had been recognized ›y Palmer and Dahle in 1922 (8). It is to be ›xpected that this effect would be even more ›evere in low-heat powders than in powders ›repared from milk heated substantially higher ›han needed for pasteurization. Freshly prepared low-heat powder can be reconstituted to ı high quality fluid milk with a taste approxinating that of fresh milk. But lacking the stabilizing effect of high heat against oxidation (4), the problem of storage stability is increased.

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However, a development in spray drying technique in which nitrogen gas is incorporated into the concentrate prior to atomization, produces a powder not only more readily dispersible than the conventionally prepared product (6) but also with a structure which, presumably, should be favorable to rapid deaeration. It consists of particles that are essentially spheres of dry foam. These particles have relatively thin cell walls and high specific surface areas—a physical structure which should facilitate the removal of oxygen prior to gas packing and, consequently, lead to greater storage stability. The increased gas permeability expected of foam spray dried milk has been confirmed and reported elsewhere (2).

This paper presents data pertaining to the flavor stability of foam spray dried milk when gas packed after prolonged deaeration in a vacuum, and when packed using an oxygen-scavenging system described by King (7). Pertinent comparative data on the effect of these packaging procedures on the oxidative stability of stored foam vacuum dried and conventionally spray dried whole milk also are included.

Experimental Procedures

Foam and conventionally spray dried whole milks were prepared from low-heat pasteurized milk (15 sec at 74-79 C) as previously described (3). Standard conditions for spray drying were 1.18-mm nozzle diameter, 327 kg of 50% total solids concentrate per hr dryer feed rate, and 127 C air temperature. Foam spray dried powders were obtained with the addition of 12.4 liters of nitrogen per kg of concentrate. Foam vacuum dried whole milks were prepared from low heat pasteurized milk (74-79 C) as previously described (10).

Immediately after manufacture, the powders to be stored without an oxygen scavenger were packed in no. 1 cans, sealed with lids in which 0.8-mm holes had been drilled through a patch of solder, and deaerated at less than 1 mm pressure. Deaeration was continued for 15 min or 18 hr, as specified in Table 1, after which the cans were filled with prepurified nitrogen (containing a maximum of 0.002% oxygen) to 0.5 atm above atmospheric pressure, and sealed.

TABLE 1

Flavor stability of various types of low-heat dried whole milk deaerated in a vacuum[a]

Type of powder	Technique for removing oxygen	No. of experi-ments	Initial flavor	Flavor score changes during storage[b]		
				2 months	4 months	6 months
Foam spray dried	18 hr evacuation—no catalyst	10[c]	36.3	−0.8	−2.3	−2.3
Conventionally spray dried	18 hr evacuation—no catalyst	10[c]	36.4	−2.2	−3.7	−3.7
Foam vacuum dried	18 hr evacuation—no catalyst	5	35.9	−0.5	−1.0	−1.1

[a] Flavor scores are averages of the number of experiments listed in Column 3.
[b] Changes relative to initial score during storage at 4 C.
[c] Each experiment was a direct comparison of powders made from the same milk concentrate and tasted at the same taste panel sessions.

Details of this technique have already been described (11). This procedure was modified for powders to be stored in an oxygen scavenging system by the inclusion of a 4-mm pellet of platinum catalyst[1] wrapped in lens paper in each can and by filling the cans with a 95-5 mixture of nitrogen-hydrogen after 15 min of vacuum deaeration.

Moisture levels of the newly canned powders were determined by the toluene-distillation method. The powders evacuated for 15 min contained approximately 3% moisture; those evacuated for 18 hr contained approximately 2% moisture.

All powders were stored at 4 C. Oxygen levels in their storage atmosphere were determined by either a modified Pepkowitz-Shirley method (9, 12) or by a paramagnetic method using the Beckman Oxygen Analyzer. The oxygen in the cans of spray dried powders packed without an oxygen scavenger was determined paramagnetically—that in all other samples by the Pepkowitz-Shirley method.

Flavor evaluations of fluid milks reconstituted from the powders were made by a trained taste panel, using a scoring system previously published (11).

Results and Discussion

Table 1 shows the relative flavor stability of different types of whole milk powder stored in nitrogen after being subjected to a long period of degassing under vacuum. The results show that foaming definitely increases the flavor stability of a low-heat spray dried whole milk which has been gas packed after deaeration for 18 hr in a vacuum. While foaming reduces the intensity of oxidation during storage, it does not eliminate it. In contrast, the greater storage stability of foam vacuum dried powder (Table 1) results from a complete absence of oxidized flavor criticisms.

The catalytic oxygen-scavenging system of King (7) was next investigated, in the hope of further reducing or eliminating the storage oxidation of low-heat foam spray dried powder. Abbot et al. (1), using this system to control oxidative deterioration in high-heat conventionally spray dried milk, found a flavor stability equal to or better than could be achieved with double gas packing. They also emphasized the saving in time and labor from substituting the catalyst for double gas packing.

The system of Abbot et al. employed a palladium catalyst and an atmosphere of 10% hydrogen and 90% nitrogen. Based on the probable requirements for hydrogen and on flammability data (5), we adopted a gas mixture of 5% hydrogen and 95% nitrogen. A number of pellet formulations, employing both platinum and palladium, were obtained from two manufacturers and tested for their ability to catalyze the removal of oxygen from the atmosphere of gas-packed foam spray dried powders. The observed differences between catalysts were considered of minor importance, as all formulations achieved oxygen levels below 0.005% within one day after packing. We selected a platinum catalyst which generally reduced the oxygen to 0.001% or less overnight. At the time of these experiments the estimated cost was 0.05 to 0.1 cent per pellet.

Table 2 demonstrates the storage stability of foam spray and vacuum dried whole milk powders packed with King's oxygen-scavenging system. The use of a catalytic oxygen scavenging system has definitely improved the flavor stability of low-heat foam spray dried powder (compared with the same type in Table 1), and the flavor scores after six months of storage are comparable with those of foam vacuum dried powder. While the final flavor score losses of 1.2 and 0.9, found in the direct com-

[1] Houdry Process and Chemical Co., P. O. Box 427, Marcus Hook, Pennsylvania.

TABLE 2

Effect of catalytic removal of oxygen on flavor stability of dried whole milks[a]

Type of powder	Technique for removing oxygen	No. of experi- ments	Initial flavor	Flavor score changes during storage[b]		
				2 months	4 months	6 months
Foam spray dried	15 min evacuation—with catalyst	3[c]	36.6	−0.8	−1.3	−1.2
Foam vacuum dried	15 min evacuation—with catalyst	3[c]	36.1	−0.1	−0.8	−0.9
Foam spray dried	15 min evacuation—with catalyst	11	36.5	−0.4	−1.7	−1.4
Foam spray dried	15 min evacuation—no catalyst	6	36.3	−0.7	−2.5	−4.6

[a] Flavor scores are the averages of the numbers of experiments listed in Column 3.

[b] Changes relative to initial score during storage at 4 C.

[c] Each experiment was a direct comparison of powders made from the same milk concentrate and tasted at the same taste panel sessions.

parison of these two types of foam dried milk, are too close to be considered significantly different, the totality of data (Tables 1 and 2) suggests a slightly greater flavor stability for the foam vacuum dried product. Oxidized flavor disappeared from the criticisms given to foam spray dried powders packed with a catalyst, as well as from all of the foam vacuum dried powders.

Not only does the catalytic system improve the flavor stability of low-heat foam spray dried powder, but does this with the period of holding under vacuum reduced from 18 hr to 15 min. It is noted (Table 2) that foam spray dried powder packed without a catalyst after only 15 min evacuation was very unstable in storage.

With one possible exception, the oxygen level in a powder's atmosphere at one week (Table 3) was indicative of its later deterioration. Thus, foam spray dried powder packed with a catalyst and foam vacuum dried powder, with or without a catalyst, had extremely low oxygen levels and, as a group, showed the greatest flavor stability, being devoid of oxidized flavor criticisms after six months of storage. Foam spray dried powder packed without a catalyst after only 15 min evacuation had the highest oxygen level and the greatest flavor deterioration. But foamed and conventionally spray dried powders packed without a catalyst after 18-hr evacuation showed a definite difference in flavor stability without a corresponding clear-cut difference in oxygen levels.

These results, however, are consistent with the data of Berlin and Pallansch (2), who found that the nature of the drying technique profoundly affected the porosity of the resultant powders. Their data indicated that 18 hr deaeration in a vacuum would effectively remove the oxygen from foam vacuum dried powder, but only incompletely from foam spray dried powder and still less completely from conventionally spray dried powder. Moreover, after storage for one week the remaining oxygen in foam spray dried powder would be in equilibrium with the powder's atmosphere, but in conventionally spray dried powder might still be in excess in the powder's interior, so that its atmospheric oxygen level would be an inadequate indication of the total oxygen available for reaction.

Regardless of the type of powder, the use of an in-pack oxygen scavenging system quickly reduces the atmospheric oxygen level to nearly zero, so that the only oxygen available for reaction is that present in the powder during the time needed for diffusing into the atmosphere surrounding the powder.

TABLE 3

Effect of dried milk type and deaeration treatment on typical oxygen levels in gas packing after one week of storage

Type of powder	Technique for removing oxygen	Oxygen level (%)	Method of analysis
Foam spray dried	18 hr evacuation—no catalyst	0.01-0.02	Paramagnetic
Conventionally spray dried	18 hr evacuation—no catalyst	0.01-0.05	Paramagnetic
Foam vacuum dried	18 hr evacuation—no catalyst	0.002	Pepkowitz-Shirley
Foam spray dried	15 hr evacuation—no catalyst	0.70-1.10	Paramagnetic
Foam spray dried	15 min evacuation—with catalyst	0.001 or less	Pepkowitz-Shirley
Foam vacuum dried	15 min evacuation—with catalyst	0.001 or less	Pepkowitz-Shirley

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